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NASA Research Grant NGR-39-008-014

INVESTIGATION OF STRESS CORROSION CRACKING OF TITANIUM ALLOYS

Semi-Annual Progress Report No. 7

for the Period

June 1, 1969 through November 30, 1969

BY

E. G. Haney

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ABSTRACT

The addition of numerous chloride salts to methanolic solutions has permitted an investigation of the effect of various cations, with valences from one to four, on the cracking of 99.5Ti, 99.0Ti, Ti-6Al-4V and Ti-13V-11Cr-3Al foil at constant Cl^- ion content. Empirical relationships were found between the water content at minimum time to failure and certain properties of the cation, namely, total ionization potential, heat of hydration and E° value. Results on Ti-6Al-4V alloy of NaCl concentration as a variable and of NaCl for pre-corrosion investigations are compared with similar work done previously with HCl additions to methanolic solutions.

Specimens of 99.5Ti foil annealed at temperatures from 1150° to 1625°F showed some differences in susceptibility to failure but the differences were not considered significant. Larger grain sizes did shift the minimum time to failure to lower water contents.

INTRODUCTION

The progress of the latest six-month period is presented in this report. A summary of the accomplishments of this program during the first two years was presented in Semi-Annual Progress Report No. 4. In the interim period (1 1/2 year) a continued investigation of the alloys, 99.5Ti, 99.0Ti, Ti-6Al-4V, Ti-5Al-5Sn-5Zr, Ti-13V-11Cr-3Al and crystal bar titanium, in methanolic solutions was undertaken. The principal investigations included studies of the following variables: inhibiting effect of water and organic additions, corrosion effects, temperature of testing solution, impurities in the solution, concentration of chlorides in solution, separation of corrosion and stress contributions to failure of specimens, electrochemical effects, and fractographic aspects. A paper covering many of the significant aspects of the work reported previously in the Semi-Annual Reports was written and presented at the 4th International Congress on Metallic Corrosion in Amsterdam in September 1969. The paper has been submitted for publication.

Research in the subsequent period has continued the investigations of the effects of chloride concentrations in methanolic solutions and of attempts to differentiate between S.C.C. (stress corrosion cracking) and corrosion effect in the failure of Ti-6Al-4V. This work was carried out with NaCl as the addition to the methanol-water solutions; the work with HCl was reported previously.

The effect of annealing temperature and grain size of 99.5Ti on cracking susceptibilities has been investigated. An extensive study on

the effect of numerous cations in methanol-water-chloride solutions on the S.C.C. of 99.0Ti, 99.5Ti, Ti-6Al-4V and Ti-13V-11Cr-3Al foil was made.

MATERIALS AND PROCEDURES

The experimental work has been accomplished with foil samples of alloys already described whose chemical compositions in weight percent are given in Table I. Table II presents the tensile properties of these same alloys in the heat-treated conditions under investigation. The ultimate tensile strength and 0.2% offset yield strength are reported in 1000 pounds per square inch, whereas the elongation is that recorded on a 2" gauge length, unless otherwise noted. Also included in Table II are the grain sizes for most of the annealed alloys, measurements being made by the linear intercept method, a minimum of 500 grains being counted per specimen in the x and y directions in the plane of the foil. The details of equipment, apparatus and testing procedures were reviewed in detail in Semi-Annual Progress Reports No. 4 and No. 6.

The effect of different cations on the cracking of titanium alloys in methanol-water solutions was investigated by adding the cation as a chloride salt, except for H^+ which was added as a water solution of HCl. The various compounds that were added are listed in Table III. Sufficient chloride was dissolved in the methanol to produce a tenth normal solution, i.e., Cl^- ion remained constant no matter what the valence of the cation. Portions of this master solution were then diluted to a hundredth normal with methanol and the desired volume

percentage of water for the testing. In adjusting the volume percentage of water in the test solution, a correction was made for any water carry-over from the water of crystallization initially present in the salt, for the water in the HCl solution, and also for the small percentage of water present in the methanol. Master solutions of unstable salts such as FeCl_2 were not prepared, rather solutions of a hundredth normal were made directly and were used within eight hours.

EXPERIMENTAL RESULTS AND DISCUSSION

Additions to Previously Reported Work -- Effect of Chloride Concentration and Effect of Pre-corrosion on S.C.C. Behavior of Ti-6Al-4V

The effect of varying concentrations of HCl in methanol-water solutions down to 10^{-6}N has already been reported^(1,2) for 99.5Ti, Ti-13V-11Cr-3Al, and Ti-6Al-4V foil. Work recently completed on Ti-6Al-4V foil with varying concentrations of NaCl in methanol-water solutions confirms that increasing the chloride ion content increases the susceptibility to S.C.C., see Figure 1. As with HCl additions the alloy is easily cracked with less than one part per million of chloride, i.e., 10^{-5}N NaCl. The quantity of water necessary to prevent cracking becomes less as the NaCl concentration is lowered and does so exponentially as seen more readily in Figure 2. The minimum time to failure is also plotted with only the 10^{-5}N NaCl point being off the curve suggesting that the exact minimum is too difficult to obtain with the existing experimental conditions because the range of water contents over which failures are observed becomes so very restricted. Under these experimental conditions

the chances of obtaining a failure at $10^{-6}N$ NaCl would appear to be practically nil. Extrapolations of the existing curves indicate failures would only be expected at $< 0.04\% H_2O$.

Pre-corrosion investigations with HCl added to methanol-water solutions have already been reported^(3,4) for 99.5Ti and Ti-6Al-4V foil. The effect of adding NaCl rather than HCl has recently been completed for the Ti-6Al-4V alloy specimens and the results are shown plotted in Figure 3 as a function of water content. The ordinate and abscissa have been normalized by dividing by the normal failure time in each solution, with the load and solution added simultaneously. If pre-corrosion has no effect on the time to failure, the results would be expected to lie along the broken horizontal line, whereas if the rate of deterioration due to corrosion proceeds independently of the applied stress, the results would be expected to lie along the broken line with a gradient of one.

The shape of the curves in Figure 3 would indicate that with shorter pre-corrosion times the corrosion mechanism predominates, especially for specimens in solutions of lowest water content and that with longer pre-corrosion times, stress is the dominate factor. The previous work on Ti-6Al-4V with 0.01N HCl added to the methanol-water solution showed much less domination of the corrosion mechanism at shorter pre-corrosion times.

Effect of Some Annealing Variables on S.C.C. of 99.5Ti

In terms of the S.C.C. of metallic systems in general it is unusual for a relatively pure metal to be cracked so readily as is commercially pure titanium in methanol-chloride environments. Annealed 99.5Ti fails by an intergranular^(3,4) mechanism suggesting that some sort of

precipitate at the grain boundaries could be involved. Also there could be a grain size dependence. If there is precipitation the extent of it presumably would depend upon the cooling rate from annealing temperature. The heat treatments employed are listed in Table II along with the tensile properties. There was no increase in strength independent of finer grain size.

The time to failure of the annealed specimens in methanol-water solutions with either 0.01N HCl or 0.01N NaCl added is illustrated in Figures 4 and 5. Failure times for all specimens fall within the same order of magnitude when the solution water content is less than inhibiting amounts; the specimens annealed at the higher temperatures were somewhat less susceptible to S.C.C. than those specimens annealed in the lower range of temperatures. Only one generalization is apparent with respect to grain size and time to failure and that is larger grain sizes tend to shift the minimum time to failure to lower water contents.

Specimens quenched in water after a 1300°F anneal (S257) were tested for tensile properties and S.C.C. behavior at various times over a period of seven months with no detectable difference in tensile properties due to the room temperature aging. The specimens exposed to methanol-water 0.01N HCl environments broke in contact with the polyethylene bottle until the last attempt made seven months after the quench. These acceptable time to failure values fell within the indicated band, see Figure 4, indicating no especial susceptibility to S.C.C.

The Effect of Cations

Having established the necessity of the presence of an appropriate anion, e.g., Cl^- , in order to crack unnotched titanium specimens in methanol-water solutions, attention was turned to the possible effects of the cation with constant Cl^- ion content. Up to this time nearly all experiments with chloride additions have been with NaCl or HCl without any significant differences in S.C.C. behavior developing as a result of the two different cations.

Now all the chlorides listed in Table III have been tried at least for 99.5Ti foil annealed at 1300°F, see Figure 6. The majority of the chlorides added to methanol-water solutions cracked the samples within times that resulted in an overlapping of points and curves so that a band was formed on the plot. This band represents curves for valences of cations from one to four. Those curves falling outside the band represent additions of 0.01N FeCl_2 , FeCl_3 or HgCl_2 which are much more aggressive in their attack. The same three exceptions can be noted for the annealed 99.0Ti foil, Figure 7. However, the resulting time to failure curves for Ti-6Al-4V foil reveals 0.01N TiCl_3 and 0.01N AlCl_3 are more aggressive than 0.01N FeCl_2 , see Figure 8. For the Ti-13V-11Cr-3Al foil even more curves no longer fit within a narrow band of values. This influence of the individual cations on S.C.C. behavior suggested that both electrochemical aspects as well as alloying of the titanium must be considered.

Let us consider first the properties of the individual ions as for instance those listed in Table IV. Comparing the total ionization potential (T.I.P.) or heat of hydration* values for NaCl or KCl with those

*The heat of hydration is defined as the heat liberated when the gaseous ion in its valence state is dissolved in water.

for FeCl_2 , FeCl_3 or HgCl_2 , it is evident that the salts which are more aggressive in their attack on titanium alloys also have the higher values of T.I.P. and heat of hydration. The best measure of this would be the position of the minimum time to failure with respect to water content of the methanol-chloride solution. At this point the overall mechanism of failure is a maximum. No relationship appears to exist between the water content as described and ionic radii or ΔF , the free energy of formation in water, see Table IV. However, Figure 10 shows a plot of the water content of the methanol-chloride solution in mole fractions at the minimum time to failure as a function of T.I.P. (bottom) or heat of hydration (top) for the 99.5Ti foil. The indicated valence of the cation is that of the compound before it is dissolved in the methanol, see Table III. Note first of all the exponential relationship that prevails for univalent and divalent cations, especially versus the T.I.P. The trivalent and tetravalent cations do not appear on this line. If these cations could be considered to be reduced to a divalent state, what would be the result.

Data are available for Cr, Ti and Sn in the divalent as well as the higher valence states, see Table IV. The T.I.P. and heat of hydration values for the three elements as divalent ions have been plotted and the points fall on the respective curves, see Figure 10, suggesting that Cr, Ti, and Sn act as divalent ions under these experimental conditions. Divalent ion data for Al are not available but it is known that under some conditions Al^{+3} ion is subject to hydrolysis in water to form complex ions, AlOH^{+2} or $\text{Al}(\text{OH})_2^+$, rather than being hydrated to $\text{Al}(\text{H}_2\text{O})_6^{+3}$. The Ferric ion is also readily hydrolyzed suggesting hydrolysis as a possible explanation

for Al^{+3} and Fe^{+3} not fitting the curves. Again consider that FeCl_3 and HgCl_2 are oxidizing agents in contrast to most of the other chlorides investigated. The point for Hg^{+2} on the heat of hydration curve does not seem to fit but it does fit the T.I.P. curve. Certainly T.I.P. values are more accurate than heat of hydration data perhaps implying that the value for the heat of hydration of Hg^{+2} should be higher than that given. Finally, there is the normality aspect, i.e., 0.01N FeCl_3 has only a third as many cations as 0.01N NaCl does. Testing the possibility that 0.03N FeCl_3 would shift the position of the minimum in the time to failure curve proved fruitless as only a minor change resulted.

The empirical relationship between the water content at the minimum time to failure and T.I.P. and heat of hydration of individual cations indicates a correlation should exist with oxidation-reduction potentials. Table V lists the values of interest as taken from Latimer⁽⁵⁾ for E° , the single electrode potential when each substance involved in the electrode reaction is at unit "activity". A plot of these values for the 99.5Ti alloy is shown in Figure 11. The points fall on a general curve except for H^+ . A similar plot for 99.0Ti alloy, Figure 12, even has the H^+ point on the curve which has about the same slope as that for the 99.5Ti alloy. Figure 13 presents the same type of data for the Ti-13V-11Cr-3Al alloy. The rounded bottom of many of the curves for this alloy presented difficulties in determining the exact minimum in the curve, see Figure 9, so the bars through some of the points on the plot represent the limits of this uncertainty. The scatter of the data is somewhat greater here but the curve for the 99.5Ti alloy fits the data very well. A similar plot for the

Ti-6Al-4V alloy gave the same general curve but the scatter was excessive and it is not reproduced here.

Three salts which were added to methanol-water solutions have not been discussed, SbCl_3 , NH_4Cl , and PdCl_2 . The 0.01N SbCl_3 containing solutions sometimes formed a white precipitate after short times, probably a reaction with the water, thus casting doubt on its equivalence with respect to the other chlorides. However, the time to failure curve for SbCl_3 does fall within the band for 99.5Ti, Figure 6, and Ti-6Al-4V, Figure 8. The similarity of NH_4^+ ion, the only radical investigated, to the alkali ions is well known and it is therefore not surprising that the effect of 0.01N NH_4Cl on the cracking of titanium alloys corresponds closely with that of LiCl , NaCl and KCl , see Figures 6, 8 and 9. The strongest oxidizing power of the chlorides added to methanol in this investigation is represented by PdCl_2 . In view of the consequent extreme of E° value (Table V), it would be expected that the water content of the minimum time to failure would be higher than any of the salts tried. Unfortunately, experimental difficulties have made it possible to obtain only an approximation of the water content value required. However, it is certain that the value would be between 0.15% to 0.6% H_2O for the 99.5Ti alloy and therefore Pd values would come closer to fitting the T.I.P. or heat of hydration curves than the E° curve. Some plating out also apparently took place because pitting corrosion was readily visible.

The fact that a relationship exists between a S.C.C. parameter and E° or heat of hydration in methanol-water-chloride solutions strongly suggests the cation functions in the water and not the methanol. In the case of univalent and divalent cations especially, hydration probably takes place.

Having observed how the properties of individual cations influence the S.C.C. behavior of titanium, let us reflect on the interaction between alloying elements in titanium and the added salts. The observation is that if a cation added to the methanolic solution corresponds with one of the alloying elements in the titanium alloy being tested, the S.C.C. attack is relatively more aggressive than would be expected for the 99.5Ti alloy. Consider first the two commercial purity titanium alloys whose major alloying element is Fe. The purer alloy, 99.5Ti, has less than half the Fe content of the 99.0Ti alloy, 0.07% and 0.19% Fe, respectively. Compare the relative amount of water necessary to inhibit the cracking, for instance, the 100 hour failure time. This figure for the HgCl_2 curves relative to the 100 hour value for the base is the same for both alloys, compare Figures 6 and 7. However this is not true of the FeCl_2 curves. The FeCl_2 curve for 99.0Ti requires relatively more water for inhibition than for 99.5Ti because it overlaps the HgCl_2 curve at 100 hour failure time. The effect of the Fe content of two alloys is also shown by comparing the 0.01N FeCl_3 curves, 99.0Ti requires over twice as much water to obtain a 100 hour failure as the 99.5Ti fail. The effect for alloys containing 3-4% Al in methanolic solutions of 0.01N AlCl_3 is even more dramatic, see Figures 8 and 9. The alloy containing chromium also is attacked more aggressively by additions of 0.01N CrCl_3 , Figure 9.

CONCLUSIONS

- 1) Very low concentrations of NaCl in methanol-water solutions were nearly as effective in cracking Ti-6Al-4V foil as similar concentrations of HCl, i.e., the alloy was susceptible to S.C.C. with only 10^{-5}N NaCl.

It took less water in the methanol to inhibit the cracking the lower the concentration of NaCl so that no cracking would be expected at 10^{-6} N NaCl with $> 0.04\%$ H_2O .

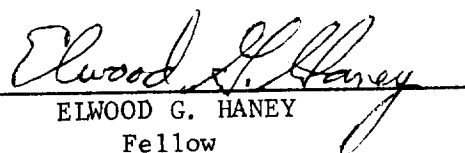
2) Pre-corrosion studies of the Ti-6Al-4V foil exposed to methanolic solutions containing 0.01N NaCl at 75% Y.S., indicate corrosion mechanisms can co-exist with stress corrosion mechanisms. The additions of NaCl tend to emphasize the corrosion mechanism more than equivalent HCl additions.

3) Various annealing experiments with 99.5Ti foil did not change the S.C.C. behavior significantly. Larger grain sizes did shift the minimum time to failure to somewhat lower water contents in methanolic solutions.

4) With a constant chloride content the effect of numerous cations was investigated for 99.5Ti, 99.0Ti, Ti-6Al-4V and Ti-13V-11Cr-3Al alloys. An empirical relationship was found to exist between the water content at the minimum time to failure and the total ionization potential, heat of hydration or E° of the cation. This implies that the cation functions in the water not the methanol.

5) If a cation added to the methanolic solution corresponds with one of the alloying elements in the titanium alloy being tested, the S.C.C. is relatively more aggressive.

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Fellow

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3. E. G. Haney and W. R. Wearmouth, Semi-Annual Progress Report No. 6, NASA Research Grant NGR-39-008-014. Carnegie-Mellon University, Mellon Institute, Pittsburgh, Pennsylvania 15213.
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5. W. M. Latimer, Oxidation Potentials, 2nd Ed., Prentice-Hall, New York, 1952.

FUTURE WORK

- 1) The dependence of cracking susceptibility on the cation of the chloride is under continued investigation, especially as a means of preventing cracking.
- 2) The behavior of foil specimens containing a tear under a continually increasing stress in methanolic solutions is still under investigation.
- 3) The fractographic studies using the scanning electron microscope are continuing.

TABLE I
Chemical Composition, Weight Percent

Alloy	Designation	Al	V	Cr	Fe	O	C	N	H
99.0Ti	Ti-75A				.19				
99.5Ti	Ti-35A				.07	.07	.023	.010	.004
6Al-4V	Ti-6Al-4V	6.4	4.0		.08		.020	.008	.013
13V-11Cr-3Al	Ti-13V-11Cr-3Al	3.2	13.8	10.6	.21	.12	.040	.030	.013

TABLE II

Tensile Properties of Foil

Alloy	Annealing Conditions	Grain Size (microns)	Y.S. (K.S.I.)	0.2% offset Y.S. (K.S.I.)	T.S. K.S.I.	Elongation, % in 2"
99.5Ti (S250)	2 hr. @ 1150°F, AC*	4.4		37.0	57.5	51.0
" (S258)	1 hr. @ 1225°F, AC	---		42.4	56.8	43.5
" (S257)	1 hr. @ 1300°F, CWQ*	---		36.6	51.0	41.4
" (S238)	1 hr. @ 1300°F, AC	15		35.7	49.9	35.3
" (S259)	1 hr. @ 1300°F, FC*	---		34.8	49.9	45.0
" (S243)	1/2 hr. @ 1500°F, AC	50		31.4	41.8	24.5
" (S248)	24 hr. @ 1590°F, AC	42		27.8	42.4	35.0
" (S247)	1 hr. @ 1625°F, AC	22		31.4	49.4	40.8
" (S245)	1 hr. @ 1625°F [†] , AC	47		26.1	42.4	30.3
99.0Ti (S270)	2 hr. @ 1300°F, AC	---		72.3	87.1	36.3
Ti-6Al-4V	---	~ 5		116.3	148.9	9.1
Ti-13V-11Cr-3Al	---	16		134.6	135.1	25.9

*AC = air cool; CWQ = cold water quench; FC = furnace cool.

[†]Specimens heated with furnace to temperature.

TABLE III

Reagent Grade Chlorides Added to Methanol-Water Solutions

Compound	Assay, %	% Water Carryover
HCl	37.8	62.2
LiCl	99.0	0
NaCl	99.9	0
KCl	99.9	0
NH ₄ Cl	99.5	0
CaCl ₂ · 2H ₂ O	78	22
SrCl ₂ · 6H ₂ O	---	40.5
MnCl ₂ · 4H ₂ O	99.4	36.4
FeCl ₂ · 4H ₂ O	100	36.2
PdCl ₂	---	0
HgCl ₂	100.0	0
AlCl ₃ · 6H ₂ O	100	44.7
TiCl ₃	---	0
CrCl ₃ · 6H ₂ O	99.5	40.5
FeCl ₃ · 6H ₂ O	100	40.0
SbCl ₃	100	0
SnCl ₄ · 5H ₂ O	100	25.7

TABLE IV

Some Properties of Common Metal Ions*

Ion	Radius	Total Ionization Potential ^a	-Heat of Hydration ^b	Free Energy of Formation in H ₂ O ^b
H ⁺	---	313.4 kcal	263 kcal	0.0 kcal
Li ⁺	0.78 A	124.3	125	-70.2
Na ⁺	0.95	118.5	100	-62.6
K ⁺	1.33	100.1	79	-67.5
Ca ²⁺	0.99	414	382	-132.2
Sr ²⁺	1.27	385	350	-133.2
Ti ²⁺	0.85	471	446	(-75.1)
Cr ²⁺	0.80	541	460	-42.1
Mn ²⁺	0.91	532	445	-54.4
Fe ²⁺	0.83	556	468	-20.3
Pd ²⁺	0.85	651	505	45.5
Sn ²⁺	1.10	506	374	-6.27
Hg ²⁺	1.12	673	441	39.4

(Continued)

TABLE IV (Continued)

Ion	Radius	Total Ionization Potential ^a	-Heat of Hydration ^b	Free Energy of Formation in H ₂ O ^b
Al ³⁺	0.57	1228	1122	-115.0
Ti ³⁺	0.64	1105	1027	(-83.6)
Cr ³⁺	0.65	1259	1105	-51.5
Mn ³⁺	0.66	1328	1098	(-19.6)
Fe ³⁺	0.67	1261	1072	-2.53
Sn ⁴⁺	0.71	2152	1827	0.65
Cl ⁻	1.81	86	85	-31.35

^aAt 0°K.^bAt 25°C.

* According to F. Basolo and R. G. Pearson, Mechanisms of Inorganic Reactions, John Wiley and Sons, New York, 1958, p. 66, or W. M. Latimer, Oxidation Potentials, 2nd Ed., Prentice-Hall, New York, 1952.

TABLE V

Oxidation-Reduction Potentials in Water*

Electrode Reaction	E° (Volts)
$\text{Li} = \text{Li}^+ + \text{e}^-$	3.045
$\text{K} = \text{K}^+ + \text{e}^-$	2.925
$\text{Sr} = \text{Sr}^{2+} + 2\text{e}^-$	2.89
$\text{Ca} = \text{Ca}^{2+} + 2\text{e}^-$	2.87
$\text{Na} = \text{Na}^+ + \text{e}^-$	2.714
$\text{Al} = \text{Al}^{3+} + 3\text{e}^-$	1.66
$\text{Ti} = \text{Ti}^{2+} + 2\text{e}^-$	1.63
$\text{Mn} = \text{Mn}^{2+} + 2\text{e}^-$	1.18
$\text{Cr} = \text{Cr}^{3+} + 3\text{e}^-$	0.74
$\text{Fe} = \text{Fe}^{2+} + 2\text{e}^-$	0.440
$\text{Cr}^{2+} = \text{Cr}^{3+} + \text{e}^-$	0.41
$\text{Ti}^{2+} = \text{Ti}^{3+} + \text{e}^-$	ca 0.37
$\text{Sn} = \text{Sn}^{2+} + 2\text{e}^-$	0.136
$\text{H}_2 = 2\text{H}^+ + 2\text{e}^-$	0.00
$\text{Sn}^{2+} = \text{Sn}^{4+} + 2\text{e}^-$	-0.15
$\text{Fe}^{2+} = \text{Fe}^{3+} + \text{e}^-$	-0.771
$\text{Hg}_2^{2+} = 2\text{Hg}^{2+} + 2\text{e}^-$	-0.920
$\text{Pd} = \text{Pd}^{2+} + 2\text{e}^-$	-0.987

*W. M. Latimer, Oxidation Potentials, 2nd Ed., Prentice Hall, New York, 1952, p. 340.

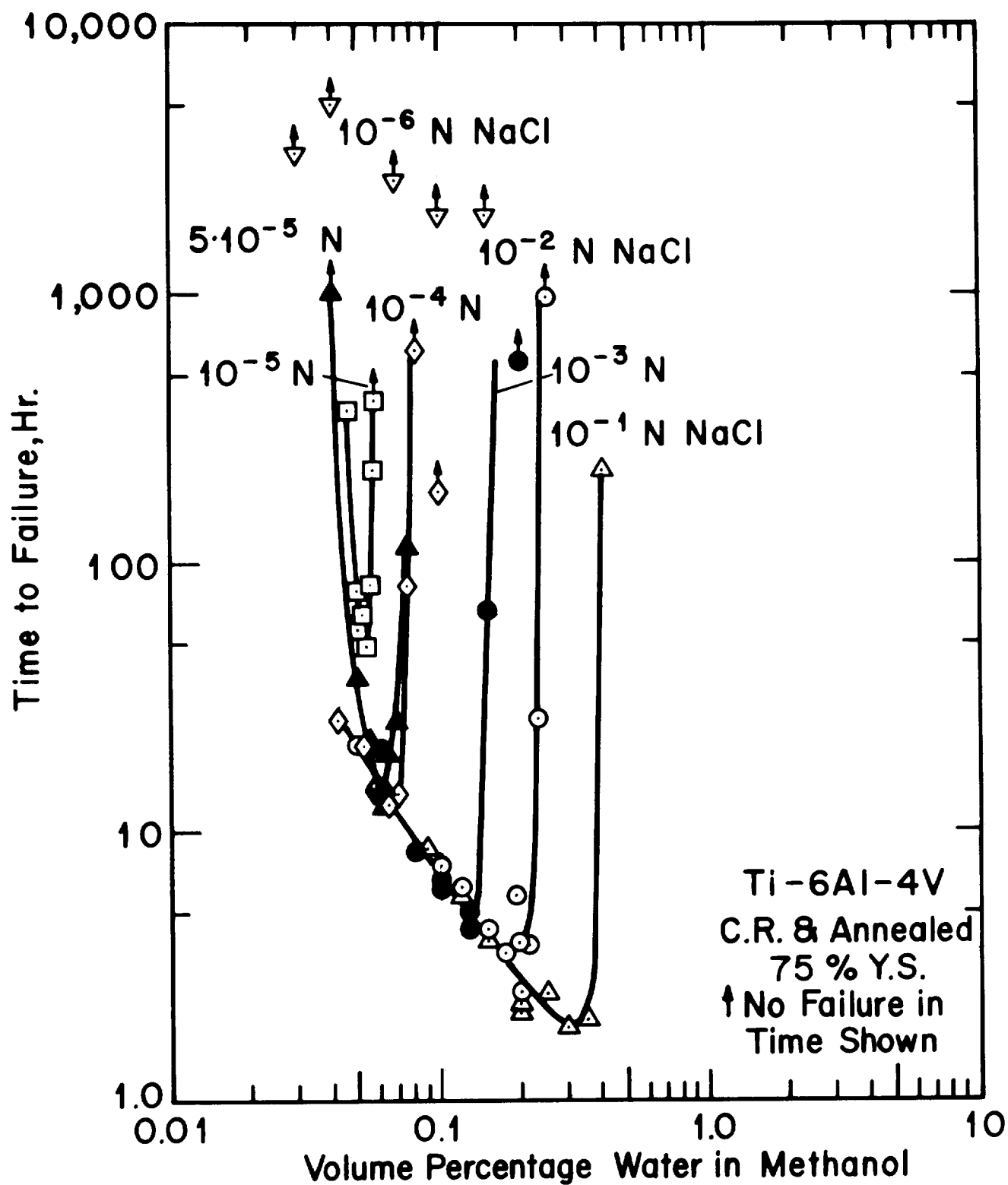


FIGURE 1. Effect of NaCl and water content on time to failure.

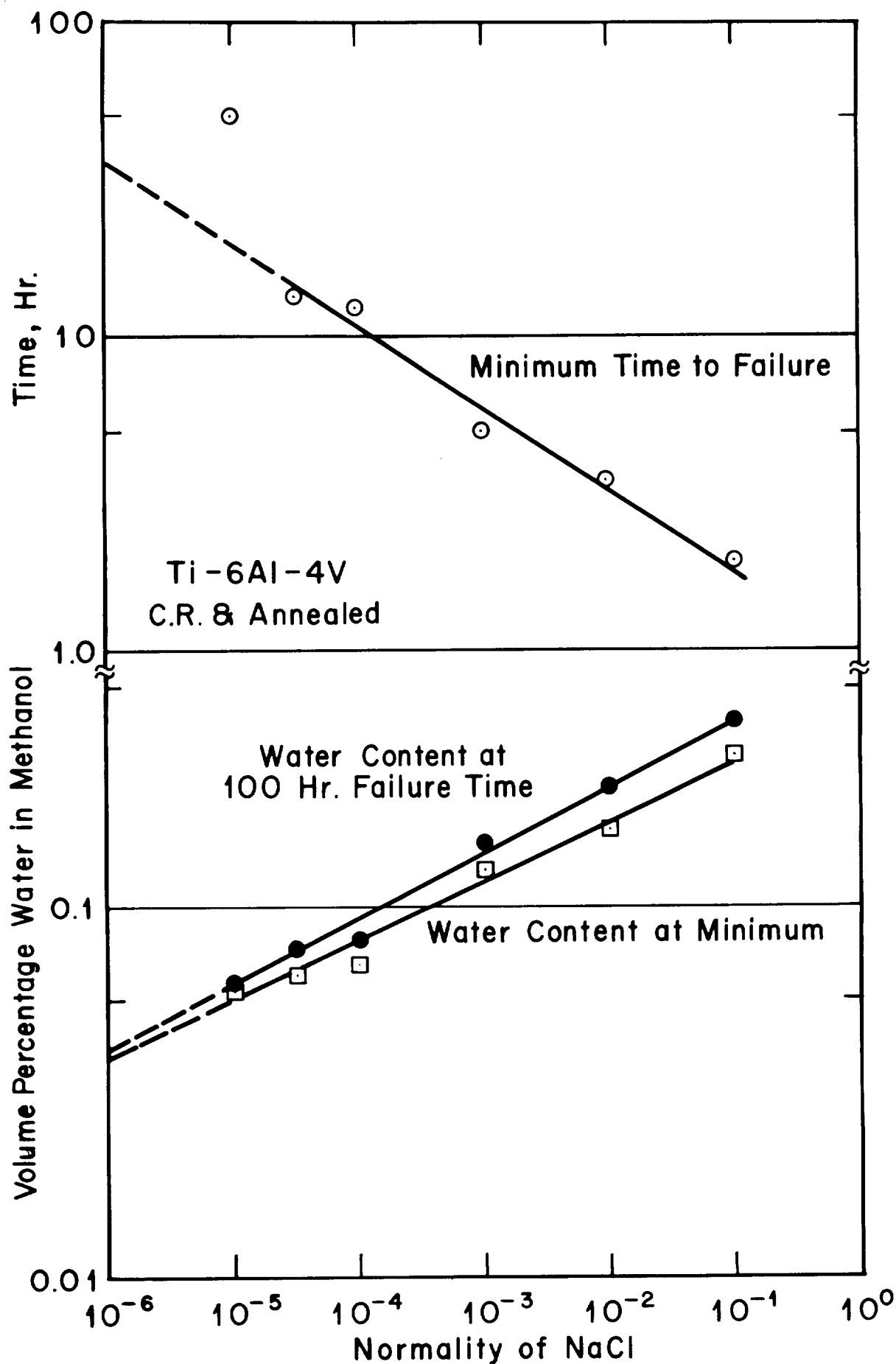


FIGURE 2. Effect of NaCl on minimum time to failure, on water content at the minimum, and on inhibition of cracking by water in methanol.

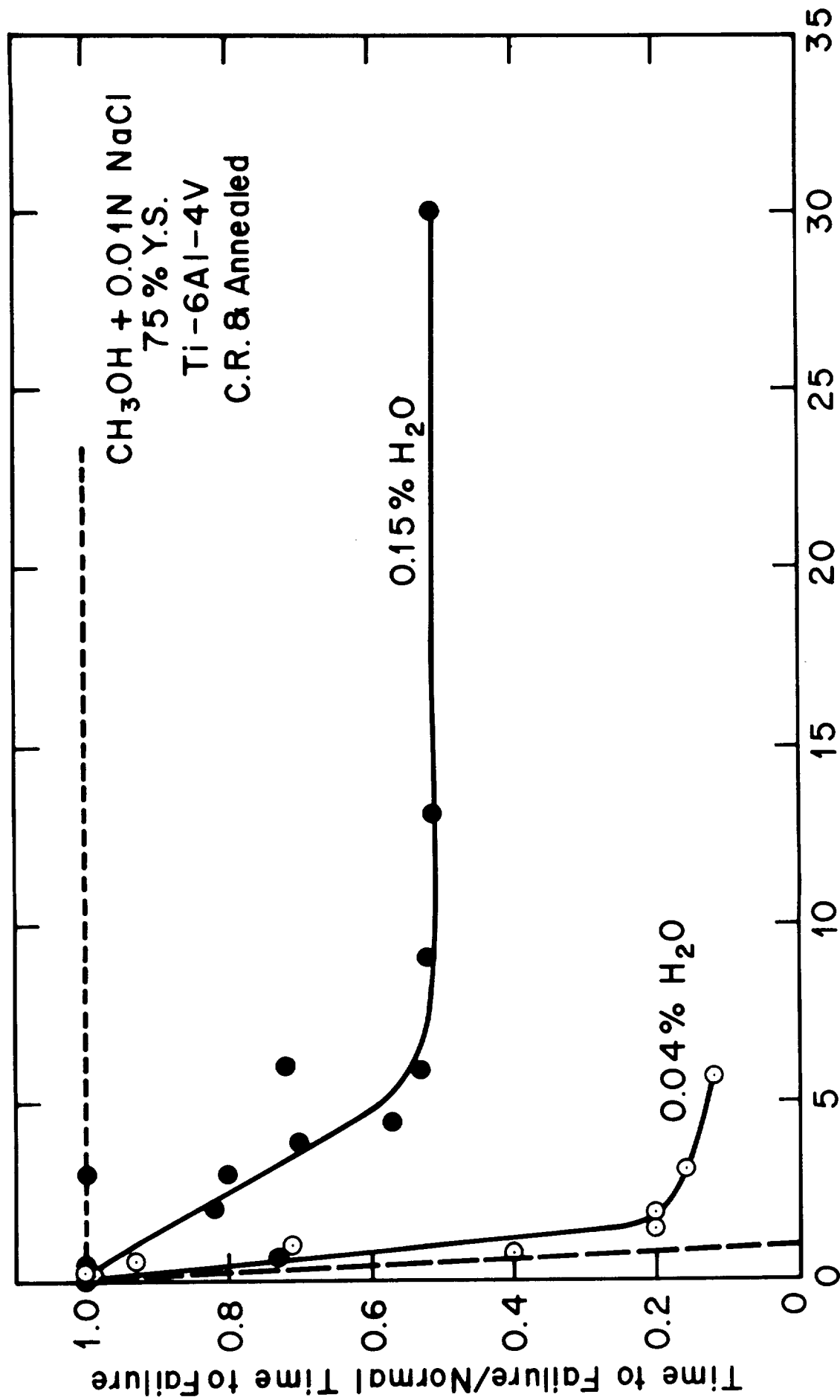


FIGURE 3. Effect of pre-corrosion on time to failure.

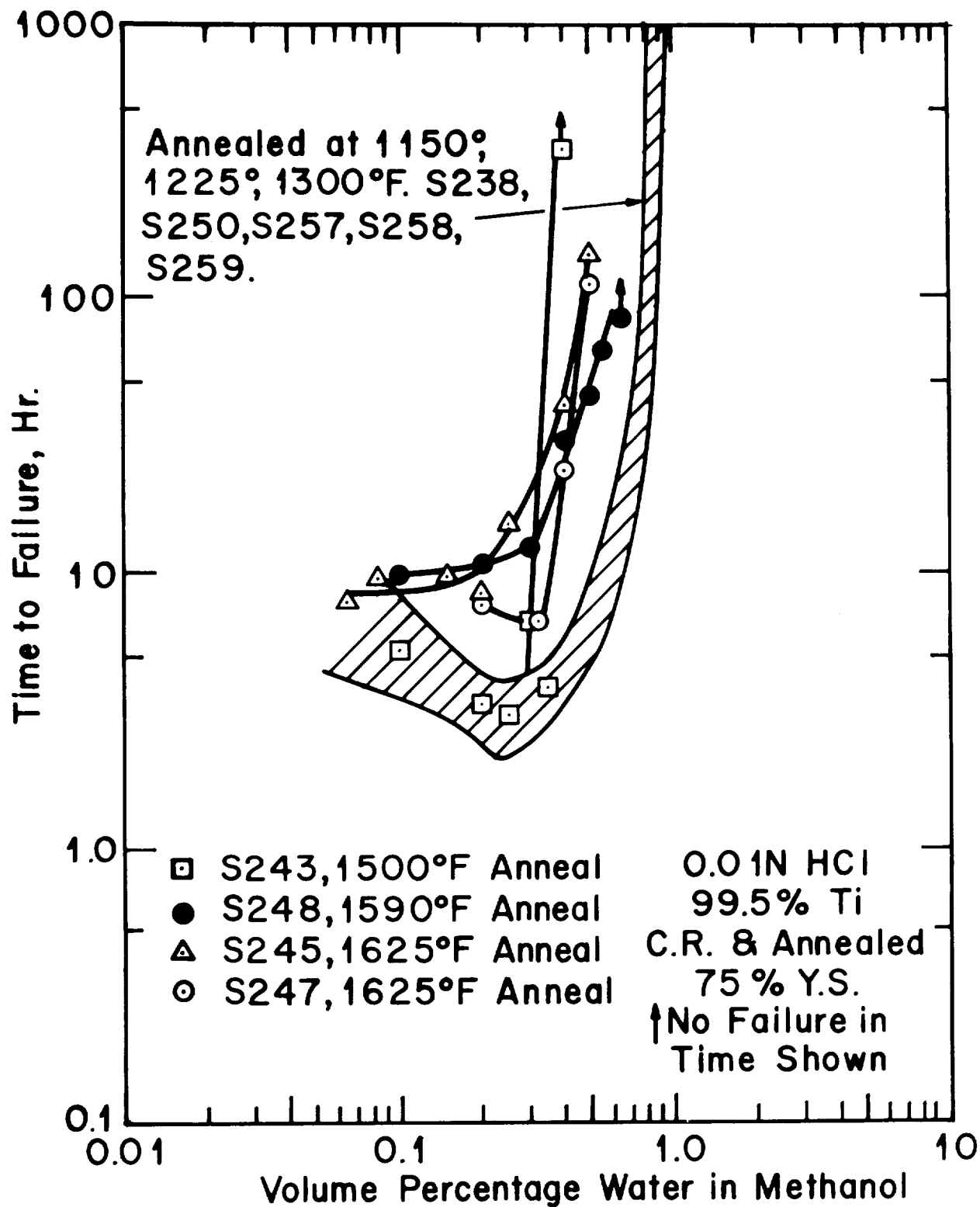


FIGURE 4. Comparison of the effect of various annealing treatments on time to failure.

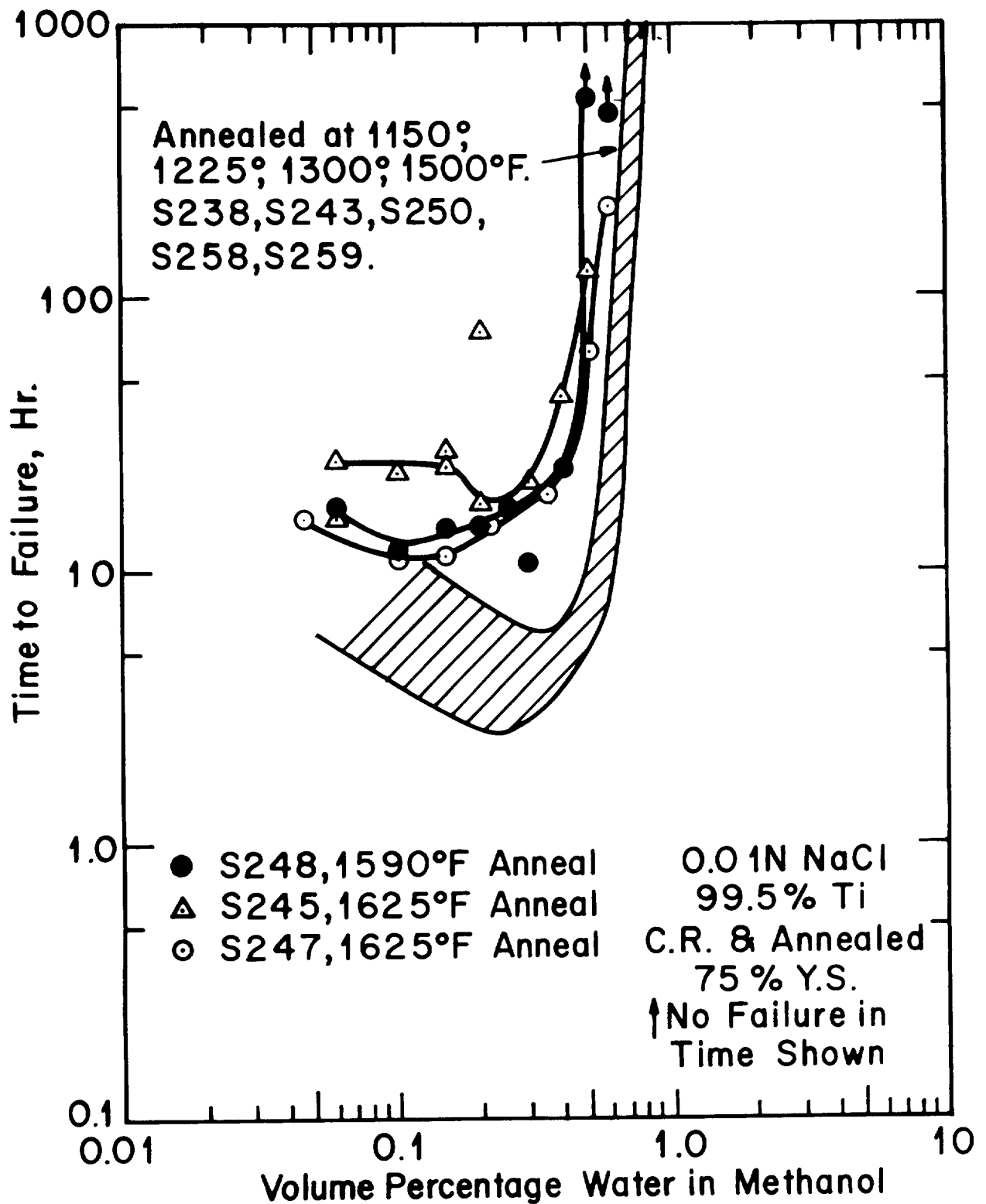


FIGURE 5. Comparison of the effect of various annealing treatments on time to failure.

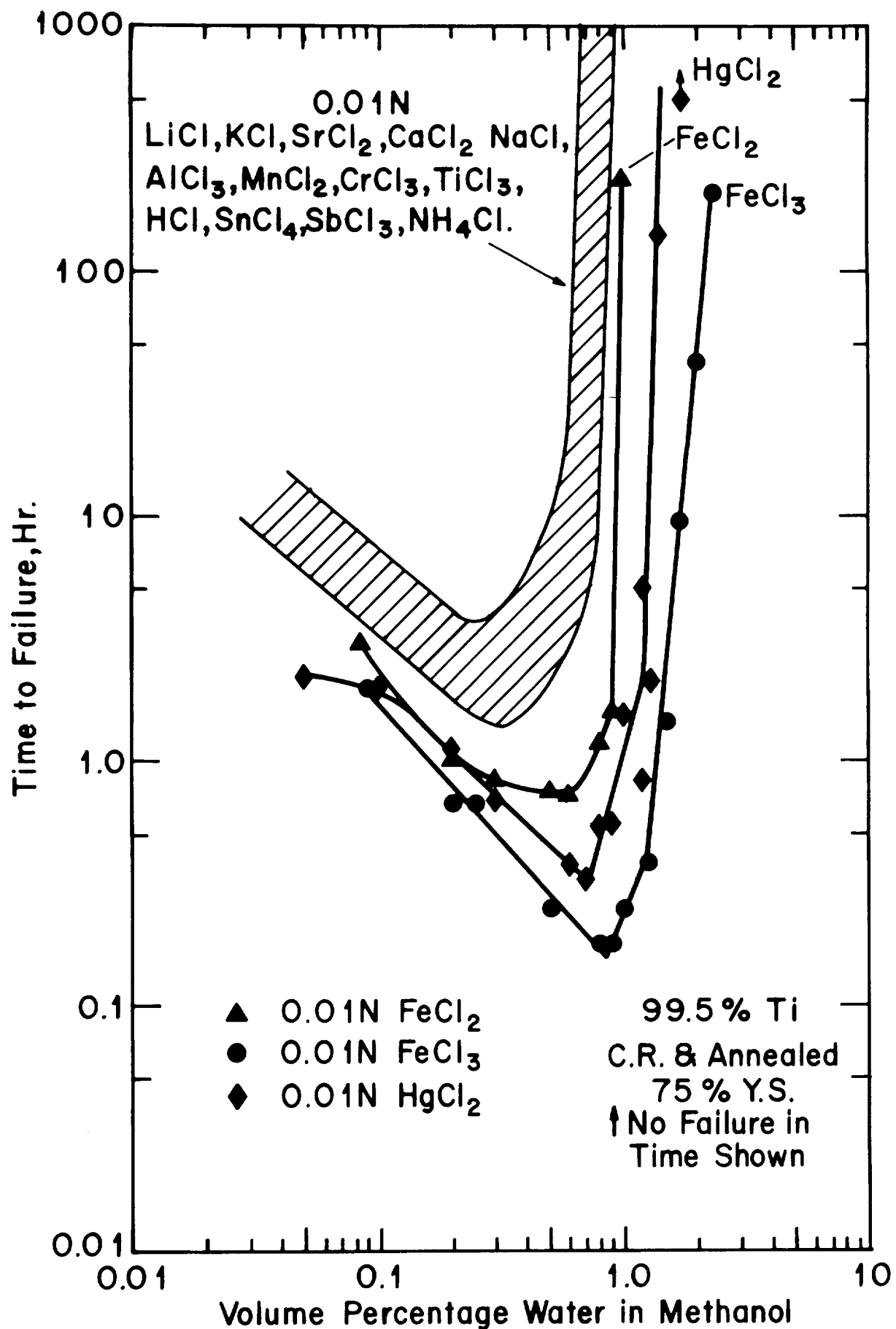


FIGURE 6. Effect of various cations on time to failure.

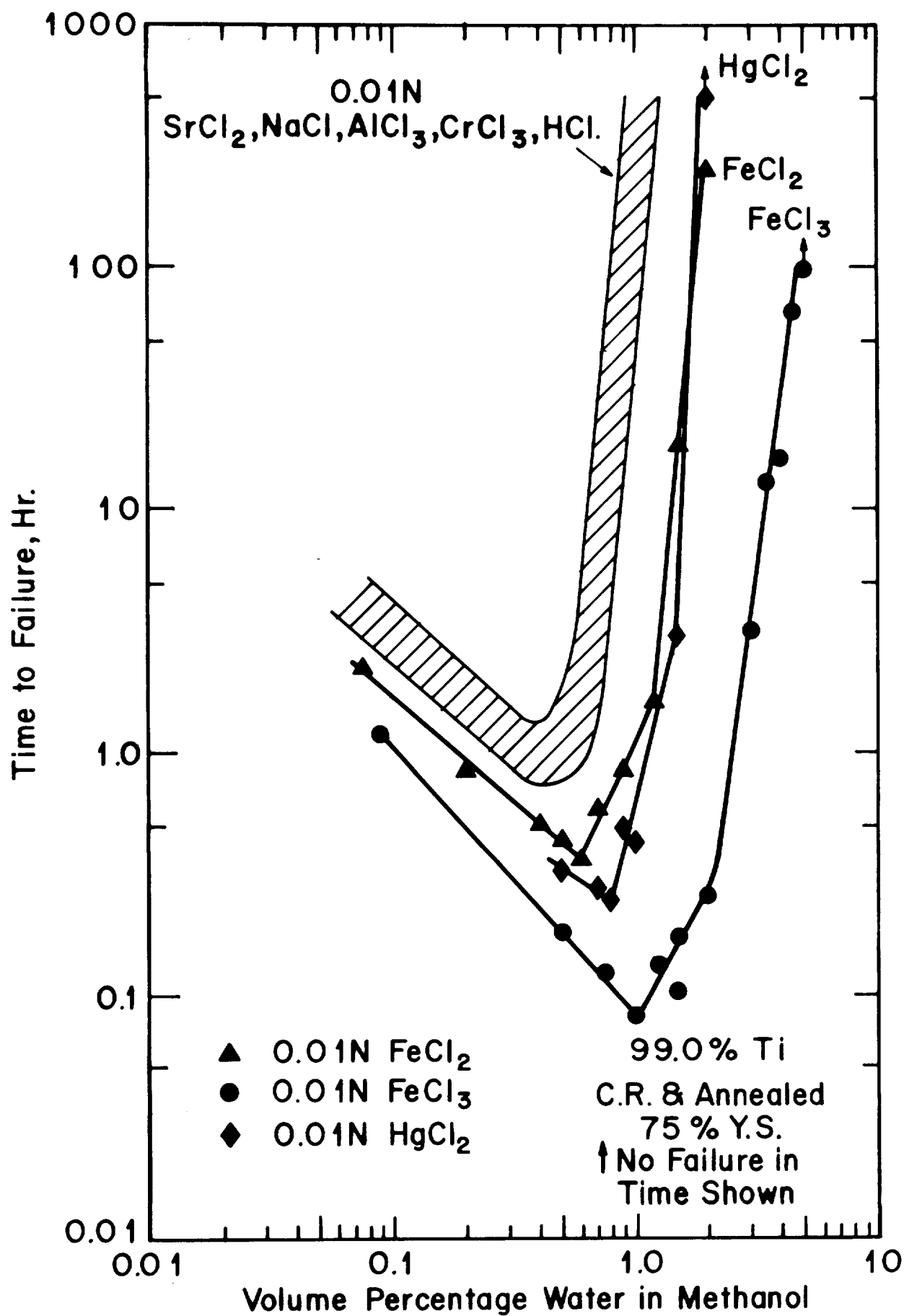


FIGURE 7. Effect of various cations on time to failure.

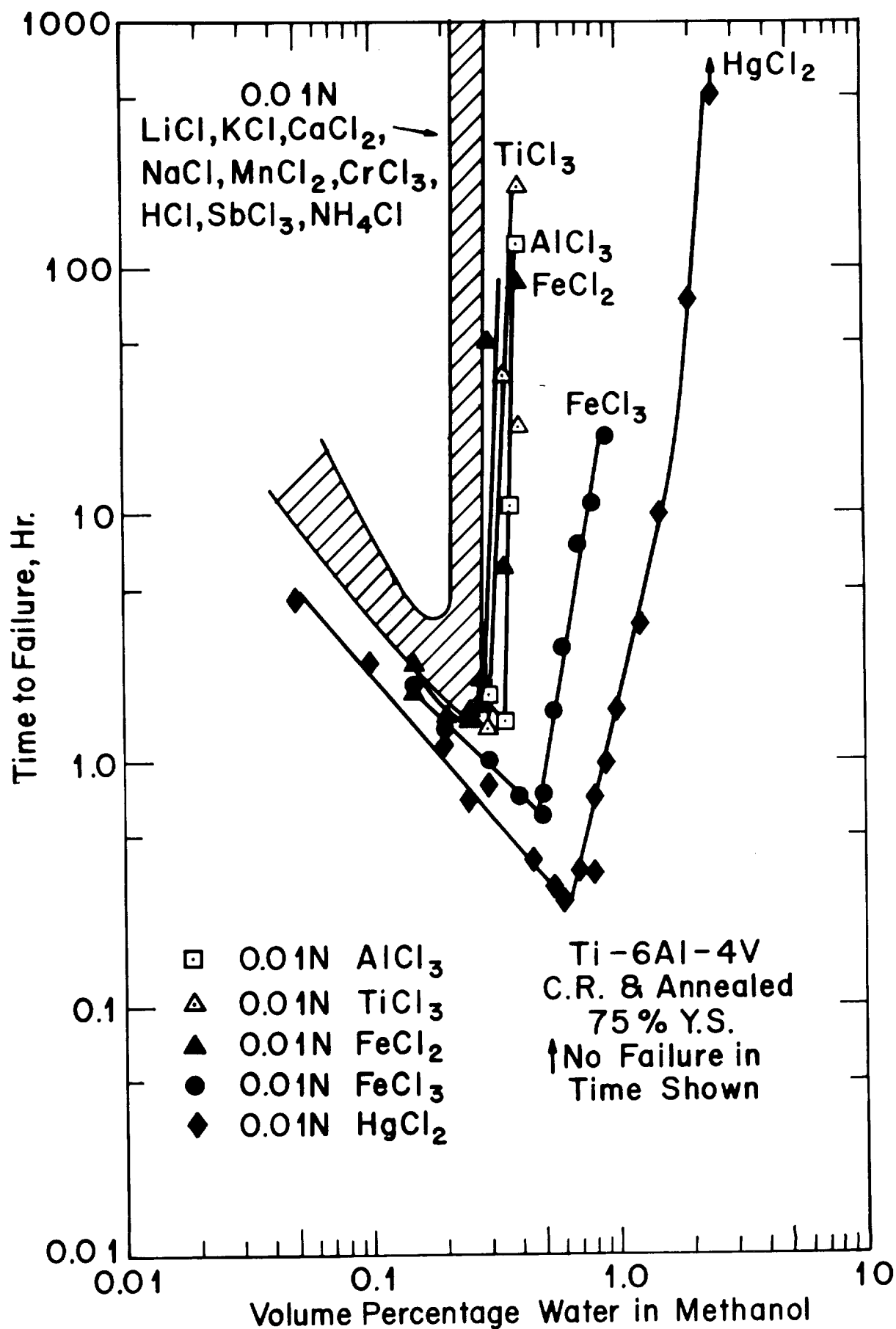


FIGURE 8. Effect of various cations on time to failure.

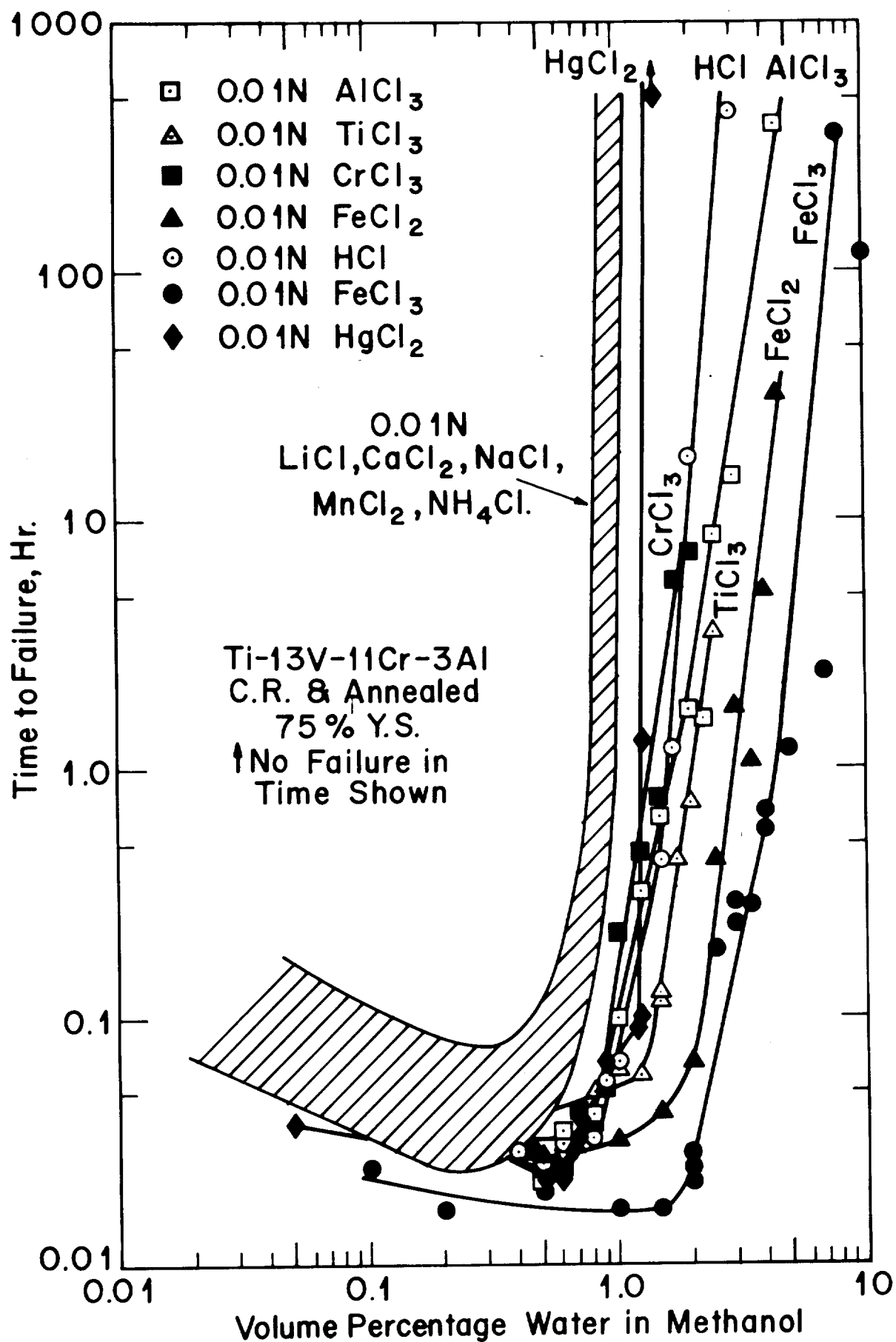


FIGURE 9. Effect of various cations on time to failure.

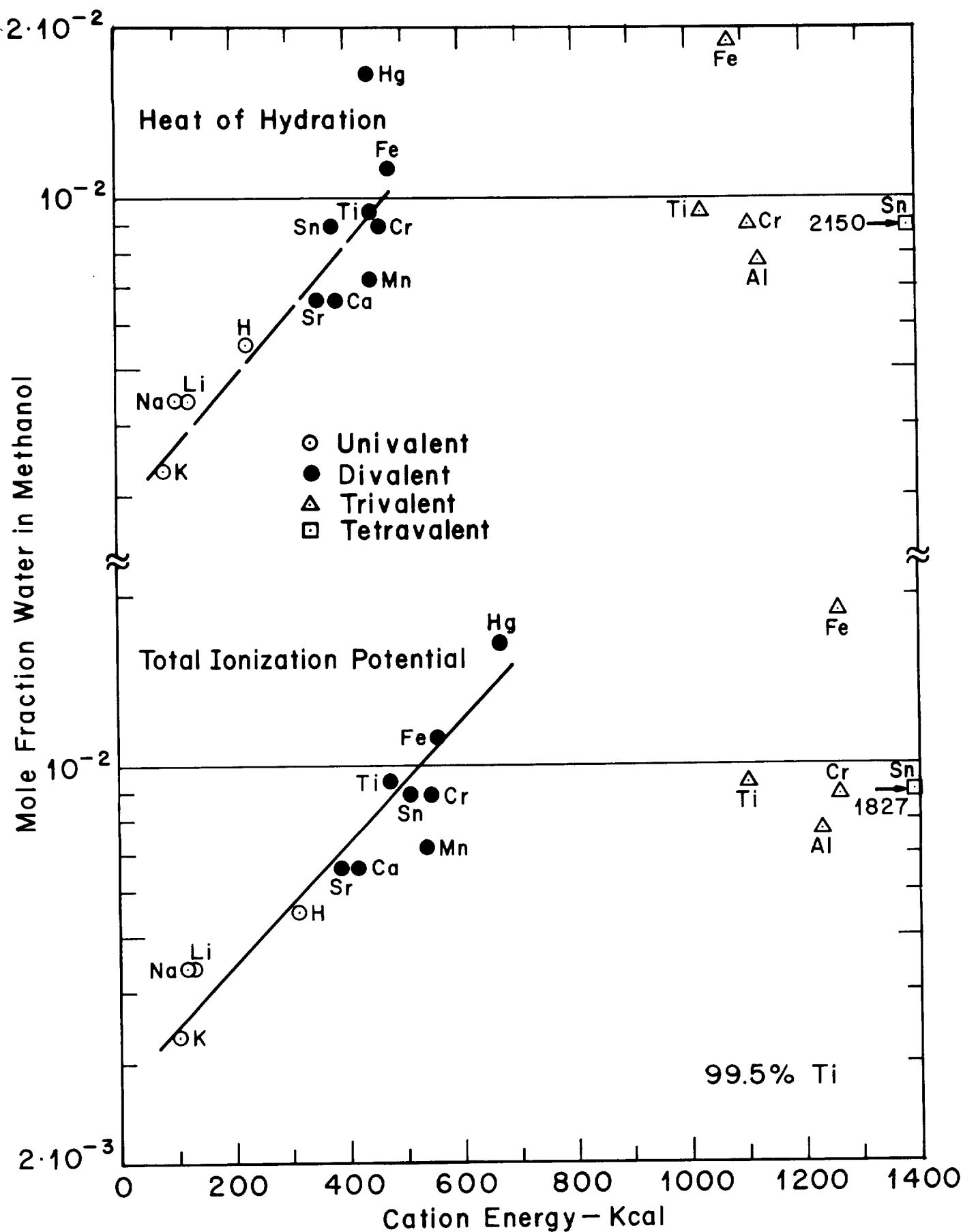


FIGURE 10. Relationship between total ionization potential or heat of hydration and the water content at the minimum time to failure.

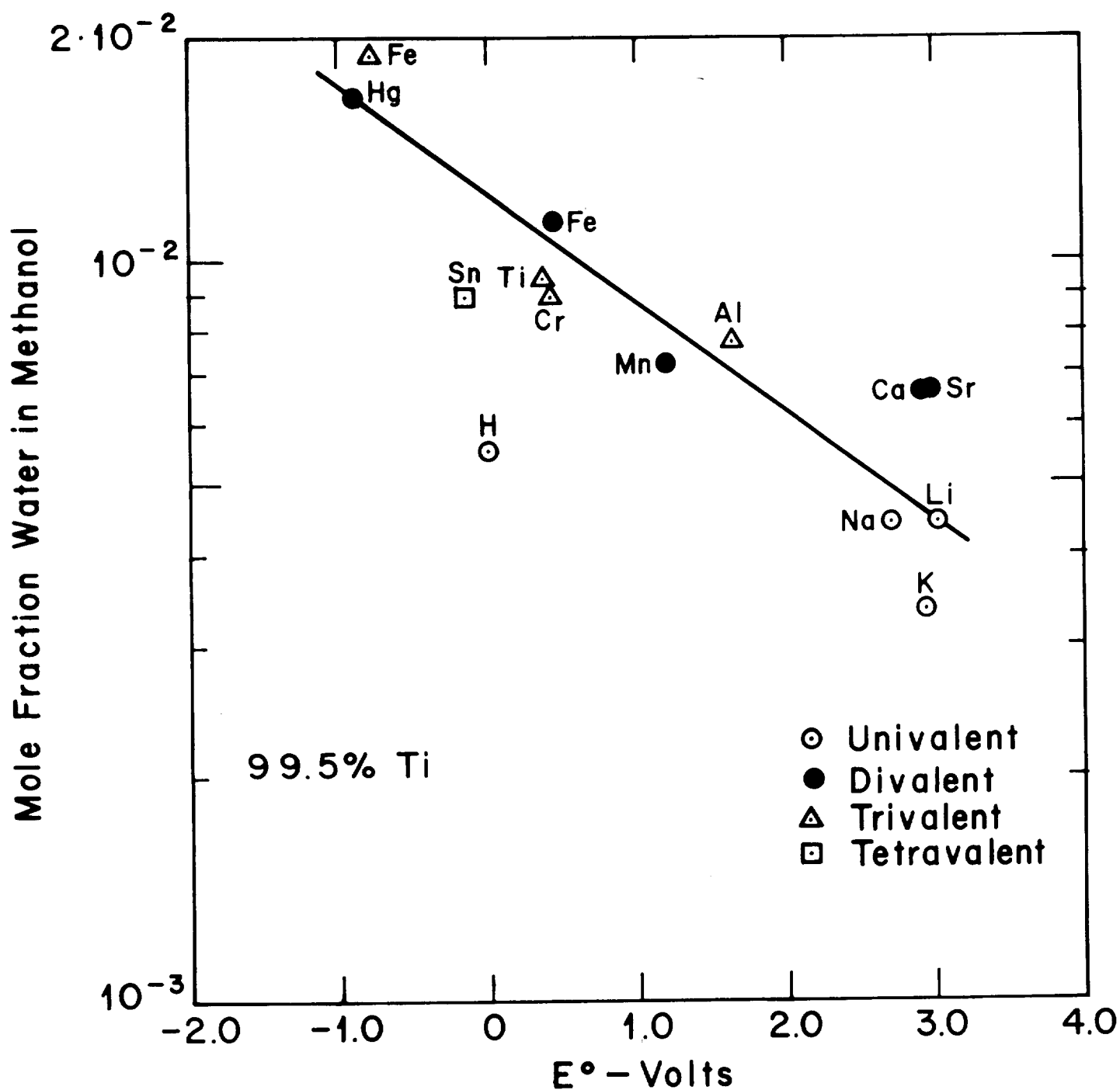


FIGURE 11. Relationship between electrode potential, E° , and water content at the minimum time to failure.

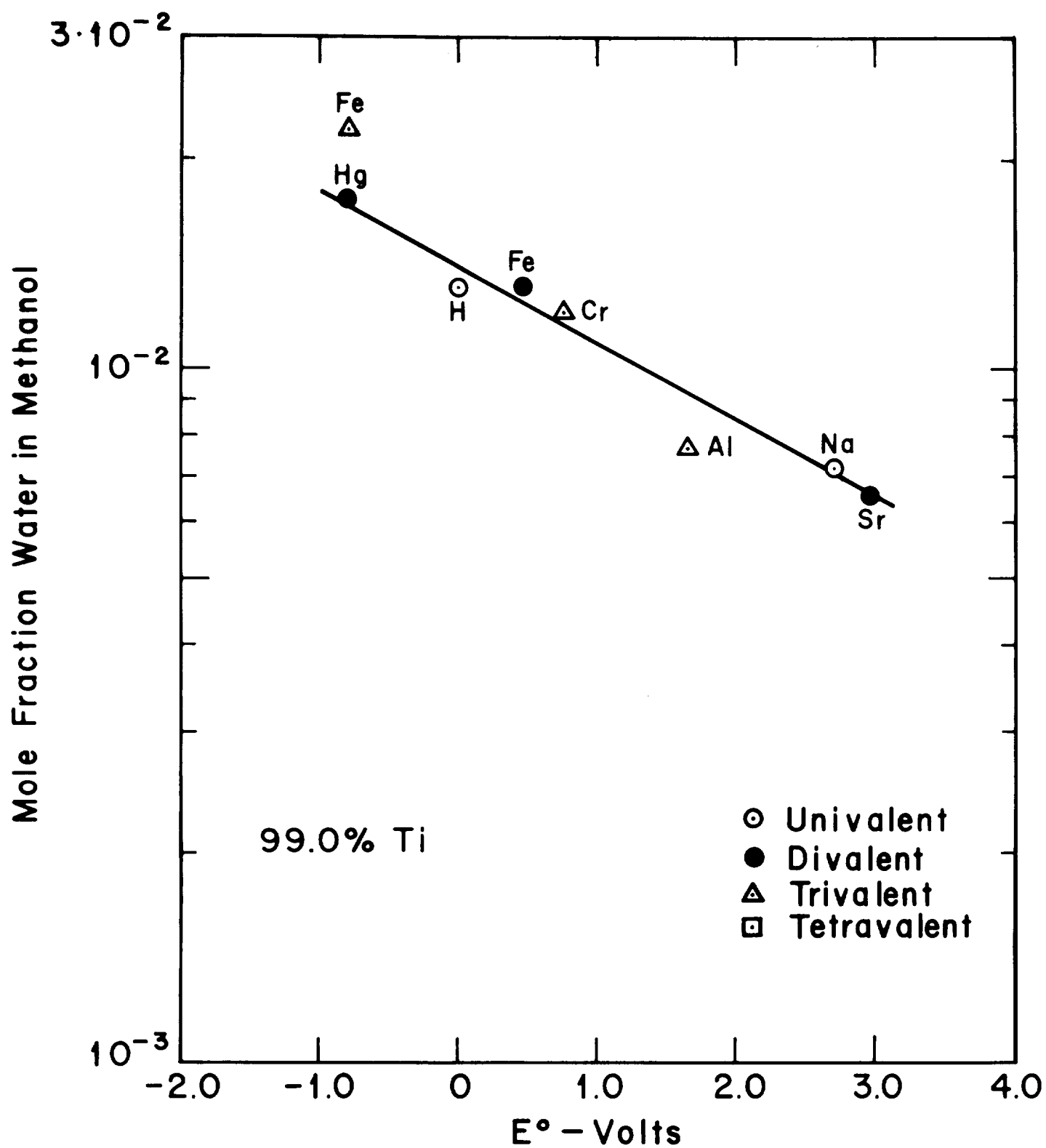


FIGURE 12. Relationship between electrode potential, E° , and water content at the minimum time to failure.

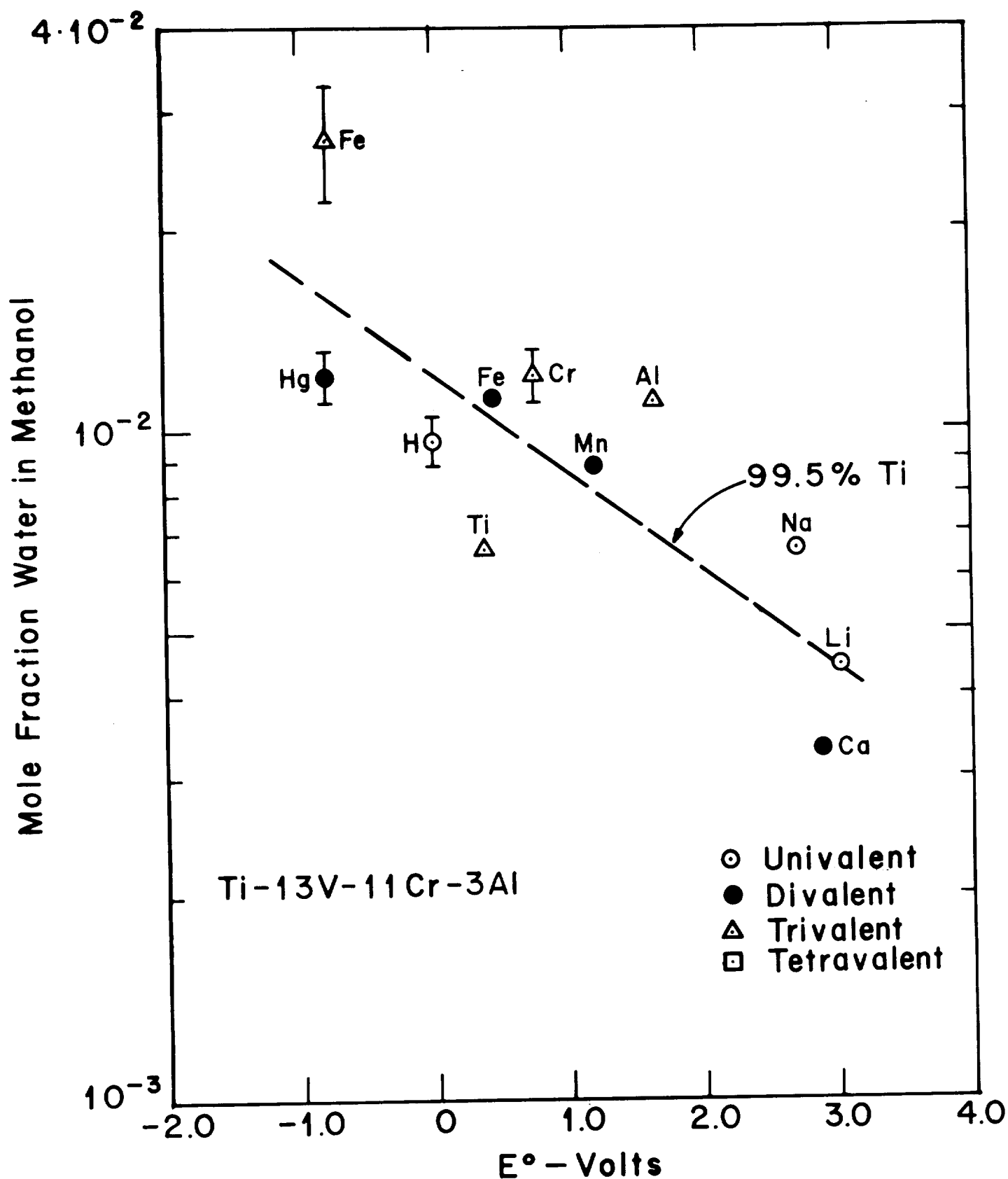


FIGURE 13. Relationship between electrode potential, E° , and water content at the minimum time to failure.

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